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April 14, 1959

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Dear Sir:

This is the first letter report under Phase III of the program being conducted under Task Order No. C, and it describes the activity during the period from February 20 to April 1, 1959.

On May 29, 1956, an effort under Task Order No. C was undertaken that was directed toward the development of an experimental hydrogen generator capable of generating enough hydrogen to provide 250 pounds of lift at sea level (about 3,500 cubic feet of hydrogen) over a period of 45 to 60 minutes. Under Phase I of this research program, a literature search was performed; an analysis was conducted of various potentially applicable systems and a selection was made of the most desirable system compatible with your specifications for the generator of interest; a laboratory investigation was conducted on the characteristics of the selected hydrogen-generating reaction; and the preliminary design of an experimental full-scale generator was prepared.

Subsequently, under Phase II, the effort was concerned with the further development and evaluation of an experimental full-scale generator based on the preliminary design prepared under Phase I. In view of the high cost of the chemicals involved, an experimental 1/5-scale generator was designed and prepared, to facilitate the investigation of the various pertinent factors involved in the hydrogen-generating reaction, and of their effects on the operation of an

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experimental full-scale generator. Based on the results of 1/10- and 1/5-scale experiments conducted in this small-scale unit, the design of the experimental full-scale generator was finalized.

After this unit was prepared by a commercial fabricator, a handling technique and an operating procedure were developed, and a full-scale experiment was carried out under field conditions on August 5, 1958. The experiment was mutually considered to have been quite successful, and served as a demonstration of the one-man operation of the experimental full-scale unit. The proceedings were recorded on movie film; a detailed review and summary of the results of, and the conclusions drawn from, the proceedings were presented in our letter dated August 21, 1958.

As a result of the full-scale demonstration, a review of the film, and several discussions, you suggested that additional consideration be given to selected areas of study in an attempt to firm up further the operation of the unit. As a result, Phase III was initiated under Task Order No. C. The objectives of this further effort are to establish the proper procedure to be used in operating the full-scale generator at low temperatures, e.g., at about 35 F; the adaptation of the procedure to operation on dry land; and the design, preparation, and installation of a pressure-relief valve in the system.

The problem in the chemical area of this program stems from the possibility, based on the available information, that, with the previously used concentration of NaBH_4 , excessively large quantities of catalyst may be required in order to achieve total generation in

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60 minutes or less at temperatures of 35 to 40 F. The resolution of this problem may lie in investigating the factors which might permit decreasing the amount of catalyst required for cold-temperature operation while still generating the required amount of hydrogen within 60 minutes.

The mechanical problems of cold-temperature generation are concerned with:

- (1) The rate of flow of the catalyst solution into the full-scale generator.
- (2) The pressure which is built up inside the unit as a result of the substantial instantaneous reaction which occurs when the catalyst solution first contacts the borohydride solution.

Experimental Activity

During this research period, three generating experiments were conducted; the 1/5-scale generator was repaired and the gas-outlet tube was scaled to that of the full-scale generator; and a pressure-relief valve was designed, fabricated, and evaluated.

Of the three experimental runs, Nos. 1 and 2 were laboratory-scale experiments designed to show the effect of catalyst distribution on the total generation time. The third run was a 1/10-scale experiment conducted at low temperature, 39 F.

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In Runs Nos. 1 and 2, the generation was followed by measuring the temperature rise. The reactions were carried out in a 10.5-inch-diameter reactor containing 2.5 liters of solution. The depth of solution in the container was approximately 2 inches. The NaBH_4 concentration was 0.56 molar, and the catalyst concentration in the whole reaction solution was 0.005 molar. In Run No. 1, the catalyst solution was added at one location at the center of the reaction pool; in Run No. 2, over the entire surface. The results of the runs were as follows:

Run No.	Initial Temperature, F	Temperature Rise, F	Reaction Time, min	Remarks
1	64	34	42	Catalyst added at center
2	66	36	50	Catalyst added all over

On the basis of these two runs, it may be tentatively concluded that there is no significant reduction in total reaction time as a consequence of distributing the catalyst solution over the surface under the conditions of these experiments. To confirm this tentative conclusion, a few additional laboratory tests of this nature will be conducted.

The experimental conditions employed in and the results obtained from Run No. 3 are as follows:

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Wt. of NaBH_4	10 pounds
Wt. of catalyst ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$)	4 pounds
Volume of river water	56 gallons
Initial temperature	39 F
Temperature rise	57 F
Complete reaction time	51 minutes
Completion of reaction	100 per cent
Hydrogen evolved	360 cubic feet
Water vapor evolved	10 cubic feet

The results of this test are encouraging. They suggest that a 40-pound limitation on the amount of catalyst that can be utilized conveniently for full-scale generation may not necessitate major changes in the generating procedures for application at low temperatures.

Also during this work period, the 1/5-scale generator was repaired to eliminate gas and water leaks. The gas outlet tube on the 1/5-scale unit was scaled to that of the full-size unit, so that the rate of gas flow from the small unit would be reduced and consequently the internal pressure from the instantaneous initial reaction would be similar to that in the full-scale unit.

In addition, effort was directed toward the development of an appropriate pressure-relief valve. For a valve of this sort to be effective in the full-scale unit, it would have to operate at low pressure, less than about 1/2 psi, and to permit a large-volume flow. Therefore, consideration was given to a simple weighted-type valve. Such a valve was prepared and its operation was satisfactory; but,

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the size of the weight needed and the necessity of mounting the valve in a near-vertical position on a fairly flexible generator unit led to distinct problems. Therefore, the weight in the experimental valve was replaced by a spring. The spring-controlled relief valve was incorporated in the top section of a T-shaped, rigid, plastic connector. One opening of the T-connector was to be inserted in the generator outlet tube, and the balloon inlet tube was to be attached to the second opening; the relief valve was located in the third opening.

The full-scale generator was set up on dry land. Approximately 560 gallons of water were added and the unit was found to be very stable. The connector was attached as described above, and the generator was filled with air until an internal pressure of approximately 1/2 psi was reached. As the pressure approached 1/2 psi, the relief valve opened and, with an increase in the volume flow, the valve fluttered and maintained a 1/2-psi internal pressure satisfactorily.

FUTURE WORK

During the next work period, a few laboratory tests will be conducted to confirm the information obtained on the effect of catalyst distribution on the total generation time. Also, by means of 1/10-scale runs, efforts will be made to establish time and temperature limits for the reaction when the catalyst weight is restricted to the equivalent of 40 pounds (for full-scale generation).

Catalyst-solution flow rates will be investigated. In addition, it may be possible, during this period, to modify the catalyst

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ring of the full-scale generator, depending on the progress made with the 1/10-scale experiments.

The total appropriation under this Task Order was \$62,579. As of April 1, 1959, the unexpended balance was approximately \$10,800.

Sincerely,

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In Duplicate

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